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# **Correlation of domain structure of TGS single crystals doped with serine with its dielectric properties – new constructed computer measuring system for quantity analysis of domain images**

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## **ABSTRACT**

This paper presents works over correlation between parameters of domain structure and pyroelectric current in IR sensors based on TGS (Triglycine sulphate). In the frames of realised works new single-crystal of TGS doped with amino acid – serine was grown. Computer measuring system making possible registration of domain structure images of non-linear dielectrics was designed and executed. We worked out measuring and analysis algorithms of domain images. Software making possible calculations and analysis of parameters of observed structures was created and static measurements of domain structure within different growth pyramids were performed. Correlation between parameters of domain structure, quantity of admixture and temperature characteristics of pyroelectric coefficient was found..

**Keywords:** ferroelectrics, pyroelectrics, domain structure, tgs

## **1. INTRODUCTION**

Process of releasing of an electric charge as a consequence of temperature changes of ferroelectric sample is well-known phenomenon called pyroelectricity. Till present days one produced many materials showing pyroelectric proprieties. One of the best material often examined and practically applied in IR detection is group of ferroelectrics with hydrogen - bond. This group of materials includes also Tryglycine Sulphate ( TGS ).

One of the most essential features of the detectors based on TGS single crystals are as follows; detectors are active elements of detection circuits where an electric response is proportional to changes of temperature, detectors show a capacitive character and high sensitivity, low noises, high speed of a detection without necessity of cooling. Additionally this material makes possible easy creation of the large detection areas at relatively low costs of a production. One of not numerous but very essential defect of this material is practically impossible repeatability of electric parameters. From this reason the works of our team were concentrated on an elaboration of method making possible fast identification of basic electric parameters of samples in the aspect of its utilisation as active elements of pyroelectric sensors.

## **2. CRYSTAL GROWTH AND DOPING**

Single crystals of TGS are grown from water solutions using a static method of the water evaporation. Pure TGS is obtained by mixing aminoacetic and sulphuric acids in proper amounts. The received solution is left for autonomous crystallisation. In order to obtain an investigative material of high purity, received crystals are taken off from the solution and after dissolving in distilled water such solution is filtered and left for renewed crystallisation. Such procedure is repeated several times. So received pure solution of TGS is admixed with a dopant in proper concentration 10% or 40%. So the received solution is put in special glass vessel in to thermostat. Here in the temperature of 35°C (ferroelectric phase) after partial vaporisation of a water on the bottom of vessel spontaneous crystallisation occurs. Small single crystals are taken off from

the solution and they become seeds for the final single crystals growth. Crystal growth takes place in temperature of 35°C in a specially designed vessel. Seed is fastened in handle, which is set in rotatory movement with the stabilised speed.

### 3. PREPARATION OF THE SAMPLES

Samples are cleaved out on (010) surface, which is perpendicular to the ferroelectric axis of a single crystal, profiting from natural cleavage plane or cut out by a wire saw, after determining of the cleavage plane. Thus obtained samples are mechanically treated in order to obtain thickness about 1mm. Polishing of samples is performed with use of fine-grained abrasive paper, during the last phase of polishing, a sample is polished on the moist blotting-paper. So prepared samples are rejuvenated by overheating to the temperature of 45°C for 24 hours. (2) Rejuvenating process is passed in order to destroy domain structure being a result of mechanical treatment. Rejuvenated samples are then left at room temperature for about 100 hours. We may assume that after 100 hours domain structure stops to change in a consequence of balance reached between free energy of the crystal and an energy of forming of the domain walls. So its structure is similar to the structure, which occurs during a normal work of the pyroelectric detector. So prepared samples are ready for an investigation of domain structure. In order to develop domain images, samples are etched using a solution of ammoniac water and then covered by the liquid crystal (3).

### 4. OBSERVATIONS OF THE DOMAIN STRUCTURE

Ferroelectric domain is an area of homogenous polarisation of the crystal. Well ordered orientation changes an optical properties of the samples' area. For that reason it is possible to observe domain structure with use of polarising microscope. In case of too little contrast of the domain images a thin layer of liquid crystal is deposited on the sample's surface. Measuring equipment (4) consists of the polarising microscope coupled with CCD camera and a computer with the frame grabbing card on the board – fig. 1.

Designed software enables the various analysis of images, including calculation of the domain amount within observed area, perimeter of a particular domain and its surface. There is also a analyses of domain images, thanks to possibility of recording of images on magnetic carrier (video tape or hard disc of the computer) possibility to investigate the processes of changes of domain structure and to make comparative

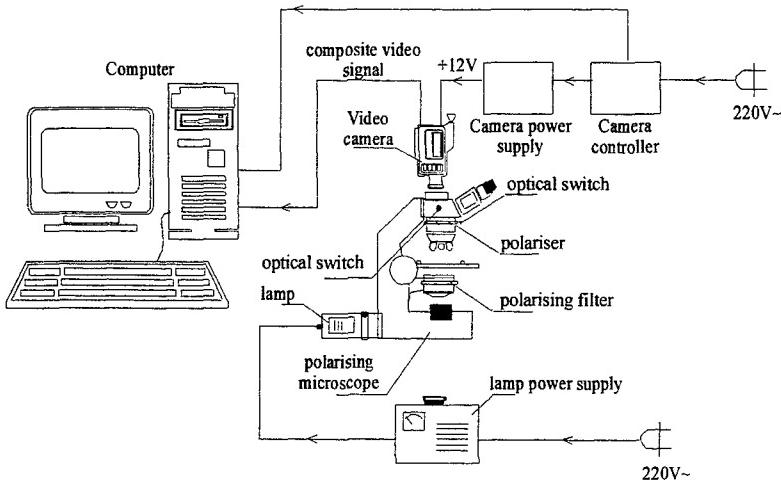


Fig. 1. Measuring system for domain structure researches

### 5. MEASURING ALGORITHM

Sample is placed in a polarising microscope. Camera records images of the domain structure in five statistically selected areas of sample's surface on its both sides. Basing on ten images registered in this manner, computer analysis of domain structure parameters was performed. Designed software makes possible registration domains' amount, its area and perimeter of domain walls.

Each area within eyeshot identified by the programme as domain was analysed and ordering parameter  $W_{sp}$  was determined for it. For each domain we measured its surface area ( $P_i$ ) and perimeter ( $O_i$ ). The ratio  $P_i/O_i$  means half of the regular circle radius ( $R/2$ ). Figure of circle was chosen because a shape of the domain influences energy of domain wall and in case of the circle this energy is minimal (circle like and lenticular domains have smallest energy). Thus parameter  $R$  may be treated as a radius of domain standardised to the circle shape. Then we calculated an area of standardised domain and the ratio between measured area  $P_i$  and calculated area. Results of the calculations for each domain are stored and on the base of it an average value of  $W_{sp}$  parameter for the whole sample is being calculated.

$$W_{sp} = 1/n \left[ \sum_1^n \left( \frac{P_i}{\pi \left( \frac{2P_i}{O_i} \right)^2} \right) \right] \quad (1)$$

Where:  $P_i$  - surface area of domain,  $O_i$  - perimeter of domain,  $N$  - quantity of domains

Basing on  $W_{sp}$  calculations, the samples are classified to one of five groups. Measurements of temperature characteristics of the pyroelectric coefficient are performed for each group.

We assumed the following boundary values of  $W_{sp}$  parameter:

Group	$W_{sp}$
p(5)	>7
p(4)	7-5
p(3)	5-3
p(2)	3-2
p(1)	2-1

Thus in the group of samples p(5) we find samples with domain structure of a large size and well developed borders, whereas in the group p(1) samples with small domains of lenticular and a circle like shapes.

## 6. MEASUREMENTS OF PYROELECTRIC COEFFICIENT

Pyroelectric coefficient is measured with use of static method, by a measurement of the pyroelectric current of short circuited sample. Sample with the attached electrodes is fastened in measuring - handle, which is then placed in a thermostat. Temperature and pyroelectric current values are registered simultaneously with the selected constant time interval.

From the theory of pyroelectrics yields, that pyroelectric effect does not step out independent. Other phenomena related with a temperature gradient within the sample take a place. The most essential disturbing factor is a piezoelectric effect. In order to eliminate this factor and other disturbances measurements of pyroelectric coefficient are conducted with very small increase of temperature and investigated sample is possibly the thinnest. An additional disturbing factor is a temperature inertia of the sample - handle set. Too fast measurements may result in deformation of the temperature characteristic of pyroelectric coefficient and its shift in the direction of higher temperatures.

### 6.1 Results of measurements

Two kinds of new ferroelectric single crystals were grown. TGS doped with 10% serine (TGSS10) and TGS doped with 40% serine (TGSS40). The spectroscopy researches confirmed a building in admixtures in single crystals' structure. We performed the described above procedures related with growing and mechanical treatment for both kinds of single crystals. Optical investigations and the electric measurements were conducted according to described measuring - algorithm.

Domain structure of TGSS10 and TGSS40 differs significantly each other (fig.1 and fig.2). An average value of Wsp parameter for all samples is 2.21 for TGSS10 and 4.63 for TGSS40. Respectively higher value of Wsp parameter the higher is difference between shape of domains and regular circles/lens. It means, that a dopant blocks process of a creation of the domain structure. This process is responsible for higher value of crystals' internal energy because process of creation of domain walls runs until depolarisation energy is in balance with energy of domain walls.

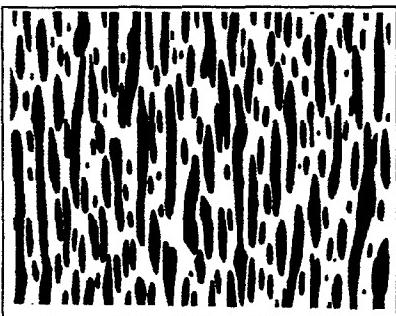


Fig. 1 Domain structure of TGSS10

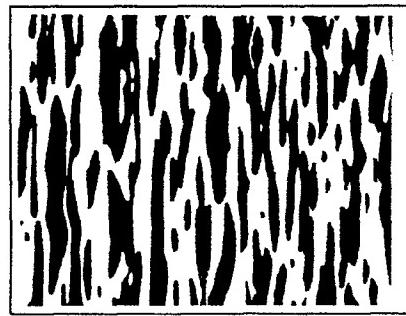


Fig. 2 Domain structure of TGSS40

Measurements of the pyroelectric coefficient showed, that samples made of TGSS10 and TGSS40 show in every group p(1)-p(5) similar values of pyroelectric coefficient and similar shapes of its characteristics fig. 3, fig. 4 - maximum of coefficient value grows together with a value of Wsp coefficient. However in case of TGSS10 width of pyroelectric peak is smaller and increase of its value is sharper within the same temperature range. Also this material shows longer linear part of characteristics of pyroelectric coefficient versus a temperature, what has an essential meaning relating an application in the detection circuits. In case of both materials basing on Wsp parameter calculation we may estimate a height of the pyroelectric peak and length of a linear part of pyroelectric current characteristics.

### 7. CONCLUSIONS

The received results encourage to the researches focused on working out algorithm of correlation of Wsp parameter with the other electric parameters of the samples, that are very essential from the point of view of its application in detectors of infrared radiation. At the present moment our works are focused on the quantitative relations between Wsp parameter, and electric parameters of the samples enabling more precise estimation of electric parameters of the samples on a base of an optical measurements.

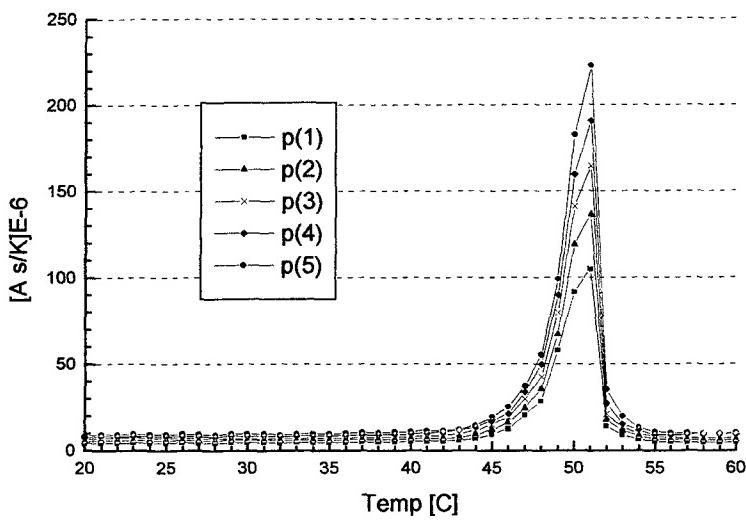


Fig. 3 Temperature characteristics of pyroelectric coefficient TGSS10

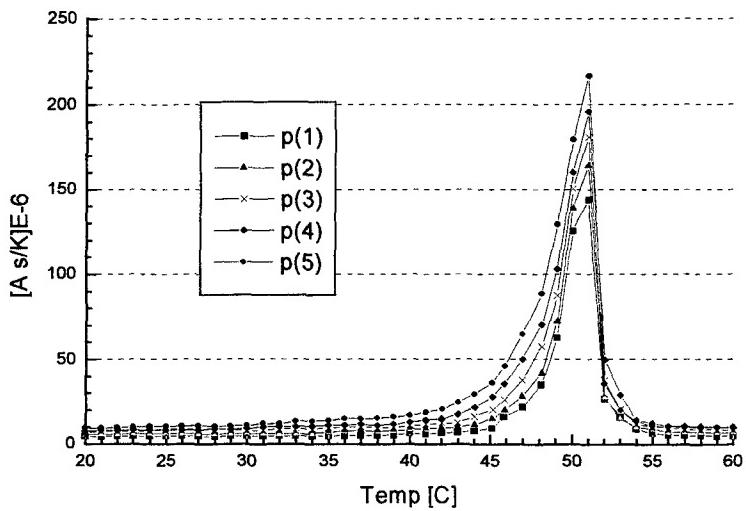


Fig. 4 Temperature characteristics of pyroelectric coefficient TGSS40

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